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IS 3242 (1989): Oxynaphthoic Acid (bon acid) [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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IS 3242 : 1989

(Reaffirmed 2000)

Indian Standard

β-OXYNAPHTHOIC ACID (BON ACID) — SPECIFICATION

(First Revision)

भारतीय मानक

β-ऑक्सीनैफथोइक एसिड (बोन एसिड) — विशिष्टि

(पहला पुनरीक्षण)

UDC 668·811 : 549·657

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

March 1990

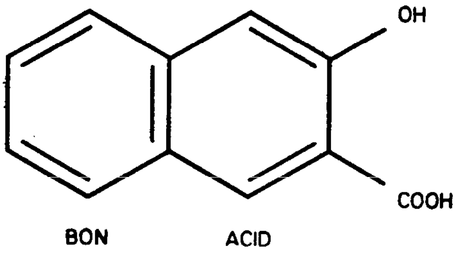
Price Group 1

Dye Intermediates Sectional Committee, PCD 11

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 13 September 1989, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

β-Oxynaphthoic acid is an important intermediate from which most of the azoic coupling components of naphthol AS (β-hydroxy-naphthoic arylamides) are derived. It is used as a coupling component for azo dyes and pigments. It is a starting material for the manufacture of 3-hydroxy-benzo-carbazole 2-carboxylic acid. It has the following structural formula:



BON ACID
(Molecular Mass 188·17)
CAS Registry Number [92-70-6]

This standard was first issued in 1965. This is being revised in order to update the requirements. In the present version thin layer chromatographic method has been introduced for determination of β-naphthol and the method for assay has been suitably modified. The requirement for ash content has been excluded and that of β-naphthol content has been updated.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

β-OXYNAPHTHOIC ACID (BON ACID) — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for β-oxynaphthoic acid.

2 REFERENCES

IS No.	*Title*
IS 915 : 1975	One-mark graduated flasks (*first revision*)
IS 1070 : 1977	Water, for general laboratory use (*second revision*)
IS 2552 : 1979	Steel drums (galvanized and ungalvanized) (*second revision*)
IS 5299 : 1969	Method of sampling and test for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of golden crystalline powder.

3.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for β-Oxynaphthoic Acid

(*Clauses* 3.2, 5.3.1, 5.3.2, 6.1 and Annex A)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. in Annex A
(1)	(2)	(3)	(4)
i)	Melting point, *Min*	217°C	A-1
ii)	Matter insoluble in sodium hydroxide solution, percent by mass, *Max*	0·2	A-2
iii)	β-naphthol content, percent by mass, *Max*	1·0	A-3
iv)	Assay, percent by mass, *Min*	97·5	A-4

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable drums (*see* IS 2552 : 1979), in fibre boards drums or polythene-lined gunny bags.

4.2 Marking

Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer;
c) Batch number; and
d) Net, gross and tare mass.

4.2.1 Each container shall, in addition, bear the minimum cautionary notice worded as under :

> 'POISONOUS; AVOID INHALATION AND CONTACT WITH SKIN; HANDLE WITH CARE'

5 SAMPLING

5.1 The representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969.

5.2 Number of Tests

5.2.1 Tests for the determination of β-naphthol content and assay, shall be conducted on each of the individual samples separately.

5.2.2 Test for the determination of remaining characteristics, namely, melting point, and solubility in sodium hydroxide solution shall be conducted on the composite samples.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to requirements of assay and β-naphthol content, if each of the individual tests results satisfies the relevant requirements given in Table 1.

5.3.2 *For Composite Samples*

For declaring the conformity of the lot to the requirements of all the other characteristics tested on the composite sample (*see* **5.2.2**), the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out as prescribed in the appropriate clauses of Annex A indicated in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Items* (i) *to* (iv)]

METHOD OF TEST FOR BON ACID

A-1 DETERMINATION OF MELTING POINT

A-1.1 Determine the melting point of the material as given in 8 of IS 5299 : 1969.

A-2 MATTER INSOLUBLE IN SODIUM HYDROXIDE SOLUTION

A-2.1 Reagents

Sodium Hydroxide Solution, approximately 10 percent (*m/v*).

A-2.2 Procedure

Weigh accurately about 5·0 g of the *prepared sample* in 500-ml beaker. Add about 300 ml water and 20 ml of 10 percent (*m/v*) sodium hydroxide solution while stirring to dissolve it completely. Filter through a weighed sintered glass crucible of porosity G4. Wash thoroughly with distilled water. Dry at 100°C, cool to room temperature and weigh.

A-2.3 Calculation

$$\text{Matter insoluble in sodium hydroxide solution, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the material taken for the test.

A-3 DETERMINATION OF β-NAPHTHOL CONTENT

The estimation of β-naphthol is done by thin layer chromatography.

A-3.1 Apparatus

Thin layer chromatographic plate, 10 × 20 cm in size, coated with silica gel G of 250 micron thickness and activated at 110°C for 30 minutes.

Micro pipette, 10 μl capacity.

Developing Chamber

Chromatographic Sprayer

A-3.2 Reagents

β *-Naphthol*, pure.

Acetone

Developing Solvent, carbon tetrachloride : glacial acetic acid (90 : 10).

Spray Solution, 0·1 percent aqueous blue B salt.

Ammonia Solution, 25 percent.

A-3.3 Procedure

Weigh accurately 1·0 g of the sample and dissolve in 50 ml acetone, dilute to 100 ml with acetone in 100-ml volumetric flask. Mix well. Weigh accurately 0·1 g of pure β-naphthol and dissolve in acetone, dilute to 100 ml with acetone in 100-ml volumetric flask and mix well. Take 10 ml of this solution and dilute to 100 ml with acetone and mix well.

Spot 10 microlitre of the sample solution and the reference solution on the thin layer plate. Place the developer in the chamber. Close the chamber with its lid and allow to achieve equilibrium. Now place the plate carefully in the chamber and allow the mobile phase to run in the ascending manner to 15 cm run from the spot at room temperature. Remove the plate from the chamber and dry off the solvent in an air drier. Neutralize acetic acid by putting the plate in ammonia atmosphere. Then couple with diazo blue B salt. After few minutes examine visually the intensity of colour of each zone developed with the sample solution and compare it with that developed with the reference solution.

A-4 ASSAY (COUPLING METHOD AFTER CORRECTION OF β-NAPHTHOL)

A-4.1 Reagents

Sodium carbonate solution, 20 percent (*m/v*).

Standard p-Chloroaniline diazo, 0·1 N.

H-Acid Indicator, 2·5 percent (*m/v*) in 20 percent (*m/v*) sodium carbonate solution.

A-4.2 Procedure

Weigh accurately about 18·8 g of sample and transfer it into a 1 000-ml beaker. Add about 200 ml water and sodium carbonate solution till the solution is alkaline to brilliant yellow indicator paper. Heat, if necessary to dissolve the material completely. Cool to room temperature and transfer it quantitatively to a one-litre volumetric flask. Dilute up to the mark with water and mix well.

Pipette out 50 ml aliquot into a one-litre beaker. Add 200 ml water and 100 ml sodium carbonate solution. Titrate while stirring mechanically at room temperature with standard *p*-chloroaniline diazo.

The end point is taken when a slight excess of diazo is shown for 3 to 5 minutes period as per the following test.

Spot a drop of coupling slurry alongside a drop of H-acid indicator solution on Whatman No. 1 filter paper, so that outspreads merge. The formation of faint red purple line at the interjunction indicates excess diazo.

A-4.3 Calculation

A-4.3.1 Percent total coupling value

$$\text{by mass, } A = \frac{V \times N \times 376}{M}$$

where

V = volume in ml of *p*-chloroaniline diazo required for titration,

N = nomality of standard *p*-choloroaniline diazo, and

M = mass in g of the sample taken for test.

A-4.3.2 Assay of BON acid,

$$\text{percent by mass} = A - (B \times 1·305\,5)$$

where

A = percent total coupling value (**A-4.3.1**). and

B = percent β-naphthol.

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BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices : Telephone

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110002 — 331 01 31, 331 13 75

Eastern : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Maniktola
CALCUTTA 700054 — 37 86 62

Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036 — 2 18 43

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